PARYLENE STICTION

Feiqiao Yu[1], Jeffrey Chun-Hui Lin[1], Po-Jui Chen[2], Yu-Chong Tai[1]
[1]California Institute of Technology, USA
[2]Robert Bosch LLC, USA

ABSTRACT

This paper presents a preliminary study into stiction between parylene C and substrate surfaces for biocompatible check-valve applications. During fabrication, parylene C is used as the structural material for the check-valve. The substrate surfaces studied include Au, Al, Si, parylene C, XeF_2 treated Si, and silicon dioxide. Stiction between different surfaces is created after sacrificial photoresist etching. Then, the stiction is measured using blister tests, and stiction mechanisms for different materials are investigated. The devices are released with different recipes to examine their effects. Finally, the results of the study reveal methods to control the cracking pressure of parylene check-valves.

INTRODUCTION

Parylene C is becoming a popular material for the fabrication of implantable MEMS (Microelectromechanical Systems) devices. Due to its biocompatibility, flexibility, and low permeability to fluids, parylene represents an ideal coating for otherwise non-biocompatible structures [5]. In addition to serving as a protective coating, parylene C has also been used in the fabrication of biocompatible check valves [2]. Such check valves are then used in implantable microfluidic flow control systems such as a glaucoma drainage valve [6]. However, since the cracking pressure of these check valves is governed by stiction between the parylene film and the underlying substrate, a comprehensive study of stiction between parylene C and different surfaces is required in order to understand, design, and create valves with specific cracking pressure and desired flow rate profiles (Fig.1).

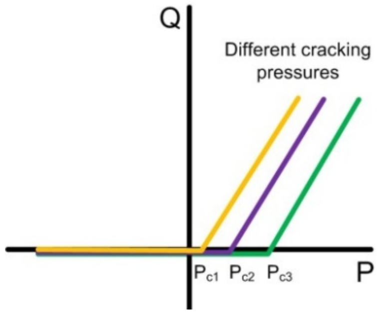


Figure 1. Theoretical flow rate vs. pressure graph of check valves with different cracking pressure

Stiction is an attraction that occurs between free standing surface micro-machined features and the substrate after the release of sacrificial photoresist [8]. Even though stiction is often an undesirable phenomenon, it can be employed to control the operation regime of thin film parylene check valves. Attempts have been made to reduce stiction for specific check valve geometries. For example, the cracking pressure of a polyimide check valve with C_4F_8/Ar non-stiction coating changed from 210kPa to 59kPa [3], and SAM (self-assembled monolayer) is also used to reduce stiction [7].

The current study presents a comprehensive investigation of stiction between parylene and a variety of different surfaces using the blister test. The surfaces under investigation include Au, Al, Si, parylene C, XeF_2 treated Si, and silicon dioxide. After quantifying surface stiction, possible mechanisms that lead to stiction between parylene and various materials are explained. In addition, different recipes for sacrificial photoresist release that may affect the resulting stiction are also experimented with. The stiction results for different surfaces under different photoresist releasing methods show that surface coating and releasing procedures used in this investigation can be used to control characteristics of parylene check valves.

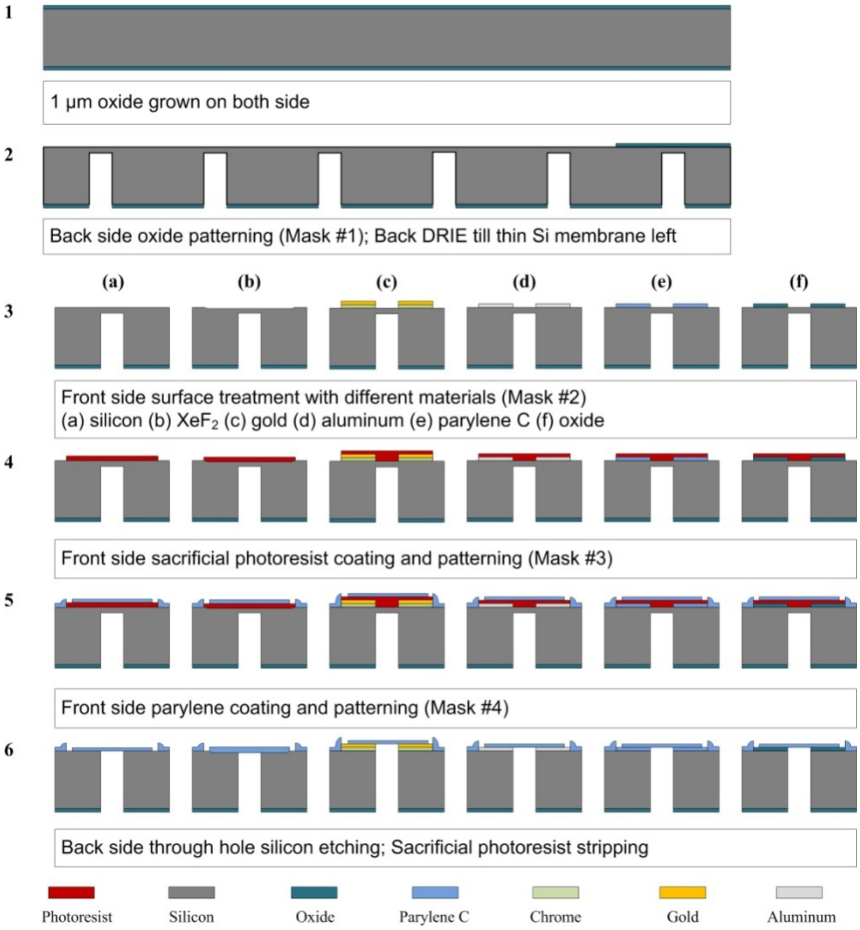
DESIGN AND FABRICATION

An outline of the fabrication procedure of stiction test devices is depicted in Fig.2. A backside circular trench 300µm in diameter is created using DRIE (deep reactive ion etching) until only a thin silicon membrane remains. Front side surface treatment is performed. These treatments include XeF_2 roughening, gold (0.2µm) and aluminum (0.25µm) deposition, parylene C coating (2.5µm), and silicon dioxide growth (1µm). After surface treatment, sacrificial photoresist and 5µm parylene layers are coated. Finally, DRIE is used again to remove the thin silicon film. Top views of the finished valves are shown in Fig.3.

After dicing the wafer, different photoresist releasing methods are used. The sacrificial photoresist of all devices is released using ST-22, after which acetone is used to remove the ST-22 residue. Then, some valves are dipped in IPA (isopropyl-alcohol) and air dried. These valves are used for surface stiction characterization. Some valves are dipped in a mixture of acetone and 5ml of

978-1-4244-5764-9/10/$26.00 ©2010 IEEE

Authorized licensed use limited to: CALIFORNIA INSTITUTE OF TECHNOLOGY. Downloaded on July 12,2010 at 18:22:34 UTC from IEEE Xplore. Restrictions apply.



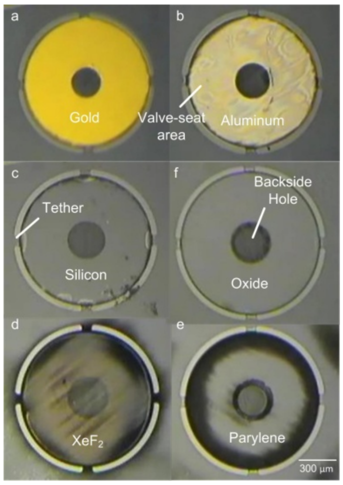


Figure 2. Top view of finished parylene check valves

Figure 3. Fabrication process for circular parylene check valve with different valve-seat surface treatments

silicone oil before air drying. These devices are used to study the effect of oil coating on parylene stiction. In addition, to test the hypothesis of stiction mechanisms, some devices are subjected a 30-second HF (hydrofluoric acid) dip followed by a quick rinse in water to remove the acid. All devices are characterized using blister test.

BLISTER TEST

Blister test is usually performed on thin films overlying a solid substrate. Through holes are used to apply pressure to the film from the back side (Fig.4). For parylene check valves, when pressure is applied, plastic deformation occurs in the parylene film that causes it to bulge to a distance d that is dependent on the Young's modulus, Poisson's ratio, geometry of the substrate opening, and thickness of the parylene film.

Due to the circular via in silicon, it can be assumed that the blister has a semispherical profile. With this assumption, stiction for can be calculated using Eqs. (1) and (2) [1].

$$p_c = \frac{3.56Et}{a^4} {d_c}^3 + \frac{4\sigma_o t}{a^2} d_c \qquad (1)$$

$$\gamma = 2.22Et\left(\frac{d_c}{a}\right)^4 + 2.00\sigma_o t\left(\frac{d_c}{a}\right)^2 \qquad (2)$$

Where p_c is the critical debonding pressure, d_c is the maximum vertical displacement of the parylene film, E is the elastic modulus of parylene C (~ 4 GPa), t is the thickness of the parylene film (3 μm), α is the radius of the blister (100 μm), and γ is the stiction between parylene and silicon. The constant σ_o represents residual stress within the parylene film. For this particular experiment where parylene is annealed at 100°C, 37.8 MPa is used as the residual stress [4]. As pressure inside the blister exceeds the critical pressure p_c, parylene film debonds from silicon. When this event occurs, p_c is used to calculate γ.

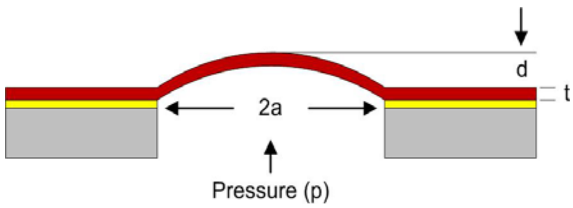


Figure 4. Theoretical blister formation during experimentation

Authorized licensed use limited to: CALIFORNIA INSTITUTE OF TECHNOLOGY. Downloaded on July 12,2010 at 18:22:34 UTC from IEEE Xplore. Restrictions apply.

During experimentation, each die is placed in a testing jig that allows fluid (N_2 gas) to apply pressure to the parylene membrane. The jig is then connected to a fluidic setup consisting of a valve, a pressure regulator, and a pressure gauge (Fig.5). The setup is placed under a microscope for observation. Pressure inside the tubing is gradually increased by adjusting the pressure regulator. The pressure gauge reads out the current pressure inside the blister. The critical pressure is recorded when debonding occurs.

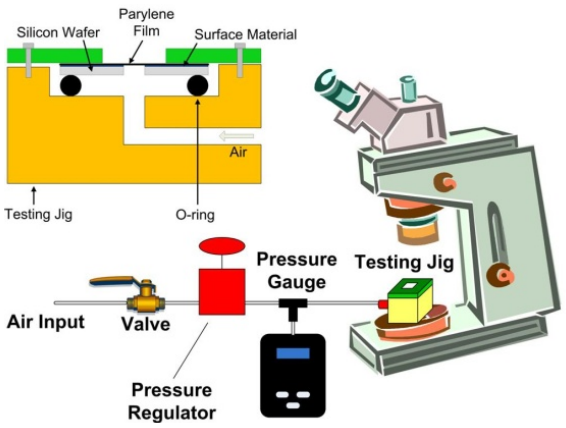


Figure 5. Stiction test setup along with a schematic diagram of the side view of the testing jig

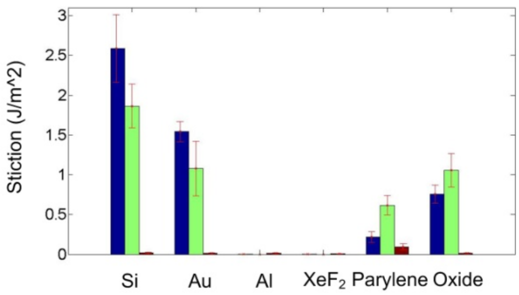


Figure 6. Stiction of parylene with different substrate surfaces after three kinds of releasing processes. Blue: acetone soak followed by IPA and water soak. Green: acetone soak followed by HF dip and water rinse. Red: soaking in mixture of acetone and silicone oil followed by direct air drying.

RESULTS AND DISCUSSION

Figure 6 illustrates stiction between parylene and different surfaces after each device is soaked in acetone, IPA, and allowed to dry in air. In addition, the average cracking pressure of the valves is also recorded (Table 1). The results show that Si has the greatest tendency to stick to parylene after drying (2.59 J/m^2). The high stiction of silicon to parylene can be explained by surface passivation. Even though silicon and parylene are inherently hydrophobic, when they are subjected to water during photoresist release, the dangling bonds on the surface of the materials tend to bond to OH^- groups in water. Such bonds make surfaces slightly hydrophilic. As the device dries, decreasing water content between the parylene film and silicon surface pulls the two surfaces together through hydrogen bonding (Fig.7). When the surfaces are extremely close from each other, Van der Waal's forces result in adhesion. Compared to Si, Au, oxide, and parylene all had gradually decreasing stiction to parylene. The decreasing values could be attributed either to increasing surface roughness or decreasing reactivity to OH^- groups. Al and XeF_2 treated Si surfaces show almost no stiction to parylene. The former result can be attributed to the high surface energy of aluminum, which precludes effective adhesion to most materials. On the other hand, XeF_2 treated Si surface display huge surface roughness. As a result, very little silicon surface actually come into contact with parylene. Thus, Van der Waal's force is not great enough to cause significant stiction.

Table 1. Cracking pressure of parylene check valves under different releasing procedures. 1: actone and IPA soak followed by air drying 2. HF dip, water rinse followed by air drying 3. soak in a mixture of acetone and silicone oil before air drying. Zero stiction means that the stiction is too small to be measured effectively

Release method	1 (J/m^2)	2 (J/m^2)	3 (mJ/m^2)
Si	2.59	1.86	18.5
Au	1.54	1.07	12.5
Al	0	0	13.2
XeF$_2$	0	0	8.2
Parylene	0.22	0.62	95.6
Oxide	0.76	1.06	13.9

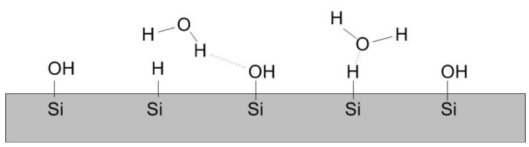


Figure 7. Hydrogen bonding that occurs between water molecules and the passivated silicon surface

Authorized licensed use limited to: CALIFORNIA INSTITUTE OF TECHNOLOGY. Downloaded on July 12,2010 at 18:22:34 UTC from IEEE Xplore. Restrictions apply.

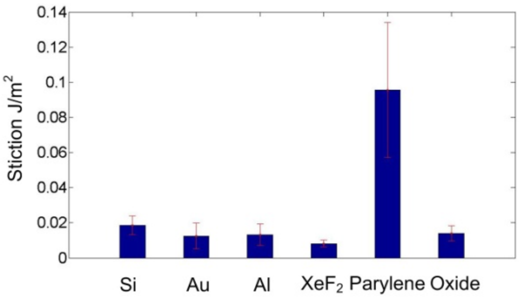


Figure 8. Stiction between parylene and various surfaces after the releasing in a mixture of acetone and silicone oil

Following the second photoresist releasing method where devices are air dried after soaking in a mixture of acetone and silicone oil, stiction between parylene and all surfaces are reduced (Fig.8). Results show that, other than parylene, all stiction values are between 0.01 and 0.02 J/m^2. These results suggest that, after silicone oil coating, stiction is no longer caused by the interaction between parylene and substrate surfaces but by the adhesion between parylene and the oil layer. Since this oil layer reduces surface passivation and the proximity between surfaces, stiction is decreased. The relatively high stiction value between two parylene surfaces can be explained by the roughness of the parylene surface, which increases its effective area to interact with silicone oil.

In order to verify the proposition that surface passivation contributes to stiction, some devices are subjected to a short HF dip before drying. This HF dip should remove some surface –OH bonds and thus reduce stiction. Results from blister tests done on these valves reveal that for certain material surfaces (ie. Si and Au), HF dip does decrease stiction slightly (Fig. 6). Even though HF dip removes much of the hydroxyl groups, the ensuing rinse in water probably introduces some –OH groups back. As a result, stiction still remains.

CONCLUSION

This study successfully quantifies stiction between thin film parylene C and various surfaces. Devices with check valve configurations were fabricated and released using different procedures. After performing blister tests, stiction values were recorded. The mechanisms that lead to stiction or the reductions thereof include surface passivation with hydroxyl groups, surface roughness, and surface proximity. Experiments show that mechanisms that can reduce the proximity between parylene and other surfaces during drying will likely reduce stiction. In addition, since different surface treatments result in different stiction, all processes and photoresist releasing methods from this study can be used to design parylene check valves with different cracking pressures.

REFERENCES

[1] M. G. Allen and S. D. Senturia, "Analysis of critical debonding pressures of stressed thin films in the blister test" *J. Adhesion ,* vol. 25, pp. 303-315, 1988

[2] P. J. Chen, D. C. Rodger, M. S. Humayun, and Y. C. Tai, "Floating-disk parylene microvalves for self-pressure-regulating flow controls" *J. Microelectromechanical Systems ,* vol. 25, pp. 1352-1361, 2008

[3] J. Han, B. Flachsbart, R. Masel, and M. A. Shannon. "Micro-fabricated membrane gas valves with a non-stiction coating deposited by C4F8/Ar plasma" *J. Micromech. Microeng. vol. 18, pp. 1-9, 2008*

[4] T. A. Harder, T. Yao, Q. He, C. Shih, and Y. C. Tai, "Residual stress in thin-film parylene-C" *Proc. MEMS*, Los Vegas, Nevada, U.S.A., Jan. 20-24, 2002. pp. 435-438

[5] W. Li, D. C. Roger, and Y. C. Tai, "Implantable RF-coiled chip packaging" *Proc. MEMS*, Tuscon, Arizona, U.S.A., January 13-17, 2008, pp. 108-111

[6] J. C. Lin, P. J. Chen, B. Yu, M. S. Humayun, and Y. C. Tai, "Minimally invasive parylene dual-valved flow drainage shunt for glaucoma implant" *Proc. MEMS*, Sorrento, Italy, January 25-29, 2009, pp. 196-199

[7] R. Maboudian, W. R. Ashurst, and C. Carraro "Self-assembled monolayers as anti-stiction coatings for MEMS: characteristics and recent developments" *Sensors and Actuators ,* vol. 82, pp. 219-223, 2000

[8] A. Witvrouw, H. Tilmans, and I. De Wolf "Materials issues in the processing, the operation and the reliability of MEMS" *Microelectronic Engineering ,* vol. 76, pp. 245-257, 2004

Authorized licensed use limited to: CALIFORNIA INSTITUTE OF TECHNOLOGY. Downloaded on July 12,2010 at 18:22:34 UTC from IEEE Xplore. Restrictions apply.